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K_Q MEASUREMENTS WITH RHEMETRICS MECHANICAL SPECTROMETER

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A testing method to measure the fracture toughness properties K_Q, which is related to a specific sample geometry and dimensions (half-inch compact tension), has been established using the Rheometrics Mechanical Spectrometer (RMS) as the loading instrument. This particular sample size was chosen so that the testing could be incorporated as part of a 50 gram evaluation scheme which was designed to provide systematic, preliminary properties information of new resins. The RMS was chosen for the experiment because of its unique (Cont'd)

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temperature capabilities. Neat specimens of Narmco-5208 Epoxy have been fracture tested at temperatures between -150°C to 250°C and interesting fracture behavior was observed. The fracture surfaces relating to these fractures were studied using Scanning Electron Microscope.

FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division, and was a joint project between WUD #43 and WUD #44. The work was initiated under Project No. 2419, "Nonmetallic and Composite Materials," Task No. 241904, Work Unit Directive 24190415, "Structural Resins" and Task No. 241902, Work Unit Directive 24190204, "Polymer Fracture". It was administered under the direction of the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with Dr. F. E. Arnold and Dr. T. E. Helminiak as the AFWAL/ML Project Scientists. Co-authors were Dr. C. Y-C. Lee, Materials Laboratory/AFWAL/MLBP, and Dr. W. B. Jones, Jr. AFWAL/MLBC.

The authors would like to thank Mr. Gary Price and Mr. Jacque Henes of University of Dayton Research Institute, for their assistance in obtaining the Scanning Electron Micrographs and part of the fracture data.

This report covers research conducted from March 1981 to December 1981.

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INTRODUCTION

Fracture toughness measurements can be distinguished from conventional tensile dog-bone testings by the fact that intentional flaws are introduced to the specimens before the testing. In tensile tests, the failure behavior shows both the material properties as well as the influence of flaws introduced during the sample fabrication process. With fracture toughness testing, the influence of the intentionally introduced flaw will overshadow the processing flaws and the fracture characteristic will be more closely related to the intrinsic properties of the material.

These two types of measurements are complimentary with each other in providing useful information for developing or selecting new resins. So it is desirable to include the fracture toughness testing as part of the 50 gram evaluation scheme which was developed in this laboratory (Reference 1) to obtain preliminary material properties information where only a limited amount of the resin is available. To bring the testing within the limitation of the 50 gram evaluation scheme, conventional specimen geometry and testing procedures have to be modified. The geometry chosen was that which has been used before by Jones (Reference 2). The sample geometry is such that the plane strain condition may not be satisfied for every material to be tested; instead, K_Q values which are related to this specific geometry can be obtained for material comparison purposes.

The Rheometrics Mechanical Spectrometer (RMS) has unique capabilities to control the sample chamber environment. The temperature range available is wide, and the heating and cooling profiles are reproducible. These features are highly desirable for measuring mechanical properties under different temperature conditions. The work described here is to develop a standardized fracture testing procedure which used the temperature capabilities of the RMS and can be suitably incorporated in the 50 gram evaluation scheme.

SECTION II

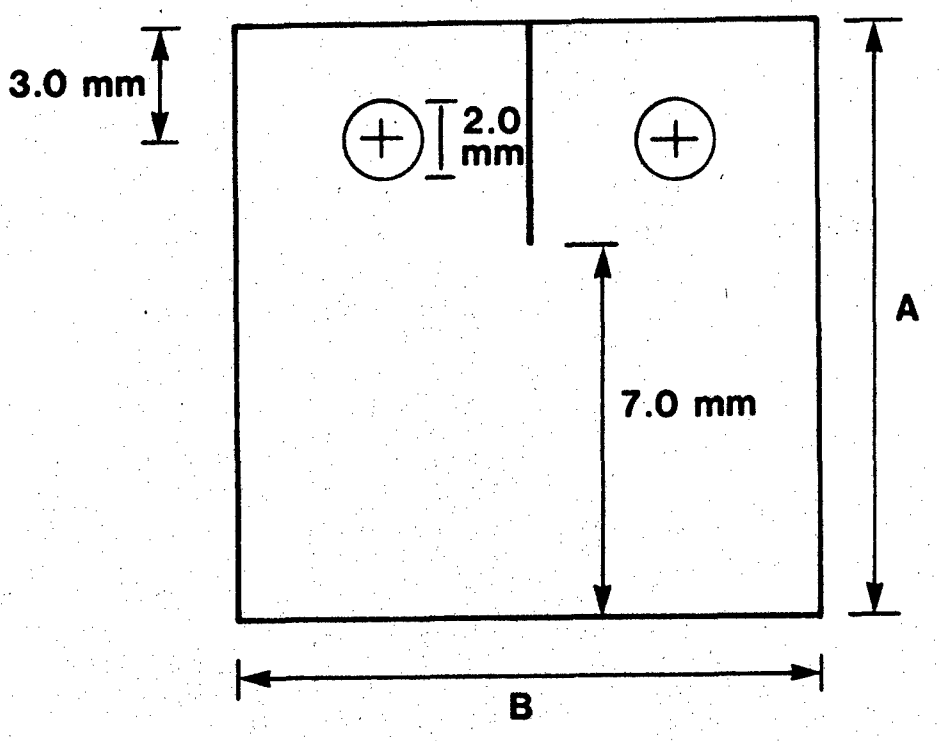
EXPERIMENTAL

The Epoxy materials used in this series of experiments are Narmco-5208 and Hercules-3501, provided by AFWAL/MLBC (Reference 3). The samples had been prepared in two different fashions. The earlier samples were prepared via the conventional method used by the Composite Branch (Reference 4). The uncured resin was poured into a stainless steel plate mold. The cured epoxy was a plate of about one foot square with thickness determined by the spacers used during the mold assembly. The plate was then sawed into half inch bars and the compact (CT) specimens were prepared from these bars.

Since part of the objective of this undertaking was to incorporate the fracture testing into the 50 gm evaluation scheme, some samples were made from silicone molds which would give half inch wide bars of 2 1/2 inches in length. As little as 3 gm of resin is sufficient to make one bar, which in turn can be used to fabricate five CT specimens. Because of the dimensions of the sample mold and CT specimens jigs, the resulted specimens from the two set of samples were different in height. The sample dimensions are listed in Figure 1. New sample jigs are being fabricated now which will change dimension B to 12mm, and keep the quantity W in the K_Q calculation (see later section) constant.

Both sets of samples were cured with the following schedule:

1 hr debulking time at 200°F
pour into mold
raise temperature 50 250°F (at 3°F/min)
10 hr hold at 250°F
remove from mold after slow cooling
raise temperature to 250°F (at 3°F/min)
raise temperature to 350°F (at 1°F/min)
hold at 350°F for 8 hr



DIMENSIONS:

PLATE MOLD

A: 13.4 mm
B: 12.75 mm
THICKNESS: 3 mm

SILICONE MOLD

A: 12.1 mm
B: 12.75 mm
THICKNESS: 2.6 mm

Figure 1. Dimensions of CT Specimens

Earlier specimens had a chevron shape saw cut. Because of reasons to be discussed in the following sections, the later specimens had a flat saw cut. All specimens now in use have a flat saw cut.

The razor blade initiated cut is a critical step in these fracture measurements. A sufficiently long fine crack must be introduced prior to the loading of the specimen with the RMS. A fresh razor blade edge is always used, and a light tap with a hammer (or another appropriately heavy object) will usually give a fine crack. Because of the specimen's dimensions, too heavy a tap will split the specimen in two halves. However, with a little practice, one can initiate cracks with the same degree of bluntness which can lead to very self-consistent, but erroneous results.

For samples that are too brittle for such tapping procedures, the specimens can be clamped in a vise, with the saw cut located just above the grips and parallel with the grip face. A fresh razor blade edge is then run over the cut with a slight downward pressure applied to the blade. This procedure can also yield satisfactory pre-cracks.

The RMS displacement convertor transforms the rotational motion of the servomotor into a vertical displacement of the upper fixture. Because of the design, only a limited range of vertical travel is allowed. In order to avoid accidental damage to the convertor by running against the limits of the travel, and to accommodate the environmental chamber enclosing the sample area, a procedure has been adopted to mount the fixtures on the RMS. The entire procedure is listed in the Appendix. The vertical displacement rate was adjusted to 0.002 in/min for most experiments. Some 0.004 in/min, 0.02 in/min and 0.04 in/min data were also obtained.

The CT specimen was mounted on the instrument through the pin-holes. The upper fixture travel was controlled by the servomotor rotational motion, which was indicated by a position control read-out. The relationship between the vertical travel of the upper fixture and the position control read-out has been calibrated. The recording of the position control can be easily converted to displacement of the fixtures. The

data were recorded with a dual-pen time base recorder. The position control and the load force output from the transducer were recorded as functions of time through the two different channels on the recorder. Instead of recording the force as a function of displacement, this mode of recording was used because it gave clear recordings of any loading and unloading cycles during the experiment. When force vs displacement diagrams are needed, they can be easily reconstructed from the original data.

The fracture toughness value, K_Q, was calculated based on the following equation (Reference 5, paragraph 9-1-4):

$$K_Q = (Pq/BW^{1/2}) \cdot f(a/W)$$

where:

$$f(a/W) = \frac{(2+a/W)\,(0.886 + 4.64a/W - 13.32a^2/W^2 + 14.72a^3/W^3 - 5/6a^4/W^4)}{(1 - a/W)^{3/2}}$$

where

Pq = Maximum load from the load diagram

B = Specimen thickness

W,a = Specimen width and crack length as defined in Figure 2.

By unloading the specimen immediately after a fracture occurred and subsequently reloading, multiple fractures can be obtained from a single specimen. The Pq value of each fracture can be easily read off from the load chart. The corresponding crack lengths a were measured afterward under a microscope.

The equation of K_Q calculation was programmed into a HP-9828A calculator. Since the relative position of the pin-holes to the top of the specimen was kept constant through the usage of a specimen jig, this distance was treated as a constant in the program. When the CT specimen dimensions were entered as input into the program, W was calculated. It is easier to measure the distance of the crack front to the end of the

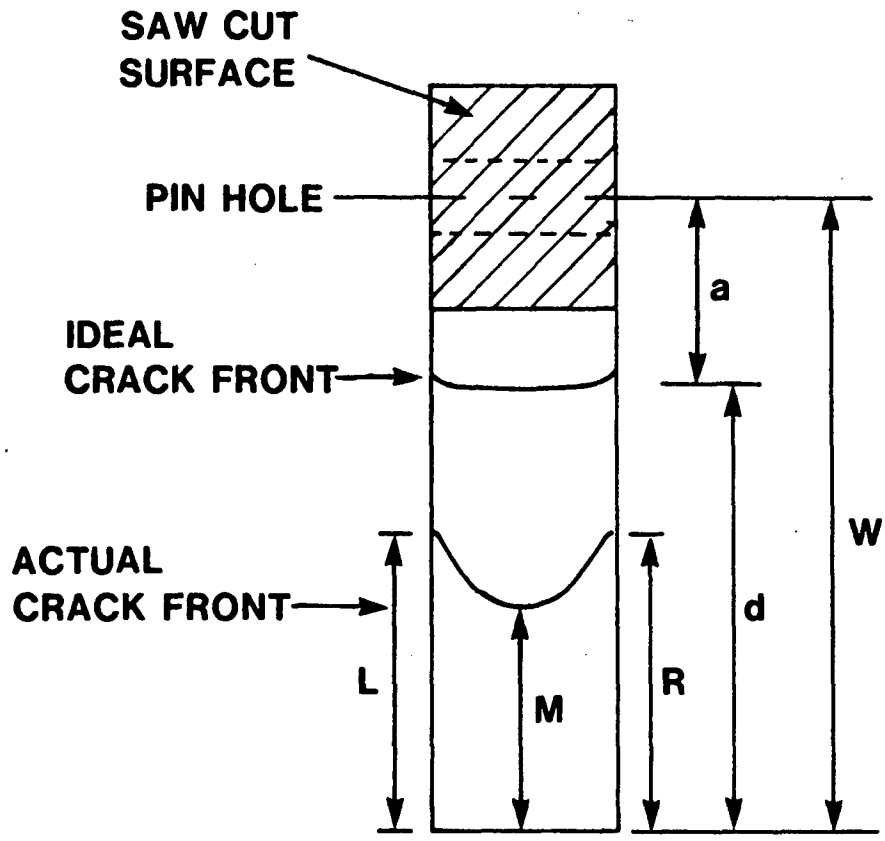


Figure 2. View of Fractured Surface of CT Specimen

specimen (d in Figure 2), so this value was also used as the input into the program. The value a and the ratio a/W were then calculated and printed as part of the output.

The American Standard Test Method (ASTM) (Reference 5) has very specific requirements for a valid crack front measurement. Few of our measurements would meet these requirements. The problem is the curvature of the crack front as shown in Figure 2. An empirical reading procedure is then adopted. Three readings were taken for every crack front from three positions, the left edge, the right edge and the tip of the crack front. They are shown as the distances L, R, and M respectively in Figure 2. Then the readings were averaged according to the equation

$$d = (L + 2M + R) / 4$$

for the calculation of a.

In the controlled temperature experiments, the chamber was preheated to the desired temperature. Then the sample was mounted with the chamber door opened for as short a time as possible. A timer was then activated. At a predetermined time the experiment was started. The procedure is designed for situations where the residence time at a temperature can change the material properties (e.g., additional cure of a partially cured system). Time to experiment initiation can then be used as an additional variable, and the time for each fracture can be calculated from the time base load diagram.

SECTION III

RESULTS AND DISCUSSIONS

1. RAZOR BLADE INITIATED CRACKS

The saw-cut slot of the earlier samples were chevron shape. To have a valid fracture for K_Q calculation, the razor blade initiated front must be beyond the chevron shaped region and be in the region traversing the thickness of the sample. Many attempts to bring this about resulted in splitting the specimens in two halves. We then attempted to initiate a small crack in the chevron region, and use the first fracture under loading to propagate the crack front into the sample thickness region. The K_Q calculation of the first fracture has to be discarded because of the position of the front. The calculated value of the first fracture is always lower than the succeeding measurements as expected.

But in too many occasions, the first fracture under loading propagated the front too far down the sample, so no valid measurements can be obtained from the specimen. Because of the invalid first fracture, it was impossible to determine if that was the result of a blunt initiated crack, though that was highly suspected as the reason.

With the straight cut, it was easier to make a sharp crack without splitting the sample. The first fracture can also be used as a valid K_Q calculation except for instances where the razor blade initiated crack was blunt. If the initiated crack was too blunt, the first K_Q value was always higher than the average and should be discarded.

The blunt crack also can cause the crack front to propagate down the specimen farther than normal. This may be due to the compliance of the fixtures and the displacement convertor. The compliance of the system will store energy during the loading, which will be released during the fracture process and be consumed in the crack propagation process. Because of the extra energy needed to initiate a blunt crack, the energy released from the system will be more and cause a deeper travel of the crack front.

Another independent study (Reference 6) at the National Bureau of Standards has also indicated that experimental results from straight cut slots are usually more consistent than those from chevron shape slots.

2. LOADING CHARACTERISTIC

It has been reported that under continuous crosshead speed loading, epoxy specimens can result in either "Stick Slip" or stable crack growth fracture (References 7, 8). However, for 5208 epoxy we have observed only one "Stick-Slip" pattern from room temperature continuous loading experiments (Figure 3a). A typical stable crack growth pattern is shown in Figure 3b. The fracture surface however was always mirror-smooth with no indication of crazing. For 3501, all load patterns tested at room temperature show the "Stick-Slip" behavior.

Figure 4 shows the Scanning Electron Micrograph (SEM) of the fracture surface that corresponds with the stable crack growth load curve shown in Figure 3b. Except with an occasional particle on the surface, which may be dust, there is no feature that suggests crazing.

The "Stick-Slip" load curve also yields a very smooth surface. However, each fracture will leave a visible crack front on the specimen surface. Examinations of the crack fronts with SEM reveal defects that are strikingly similar. Figure 5 shows the SEM taken from the specimen that gave the "Stick-Slip" pattern.

Figure 5a shows the crack front. There are circular domes along the crack front. Closer examination (Figure 5b) indicates that they may be formed from localized internal swelling after the fracture because the feature of the crack front is being smeared out.

It is interesting to note that such features have been observed before, but on quite a different scale (Reference 9). In those instances, 5208 epoxy bars were used to run torsional experiments, and the specimens were subjected to temperature scans of up to 280°C. Similar shape blisterings can be found on the surfaces of the specimen except they are

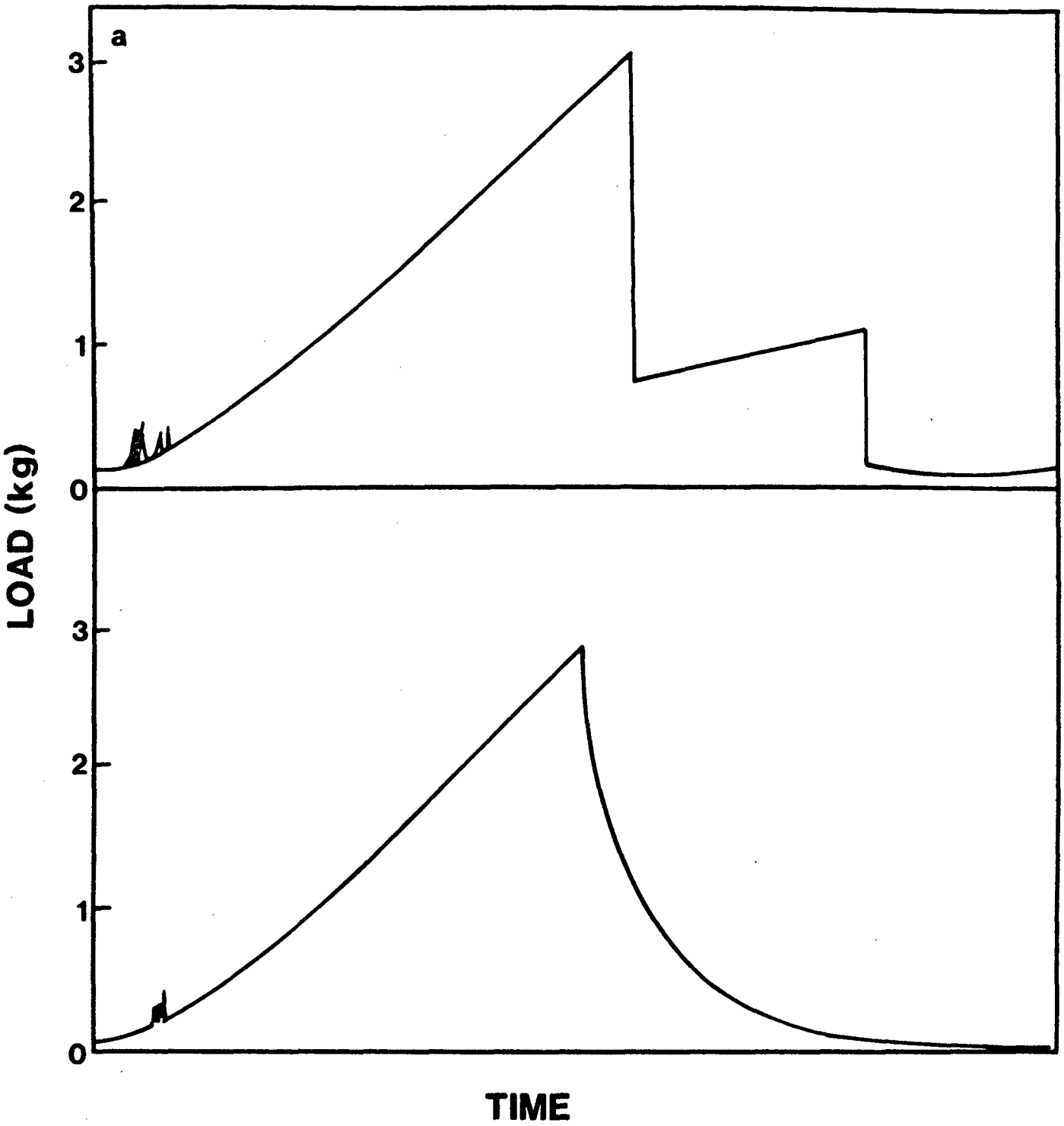


Figure 3. Load Diagrams of Slip-Stick Pattern and Stable Crack Growth Pattern

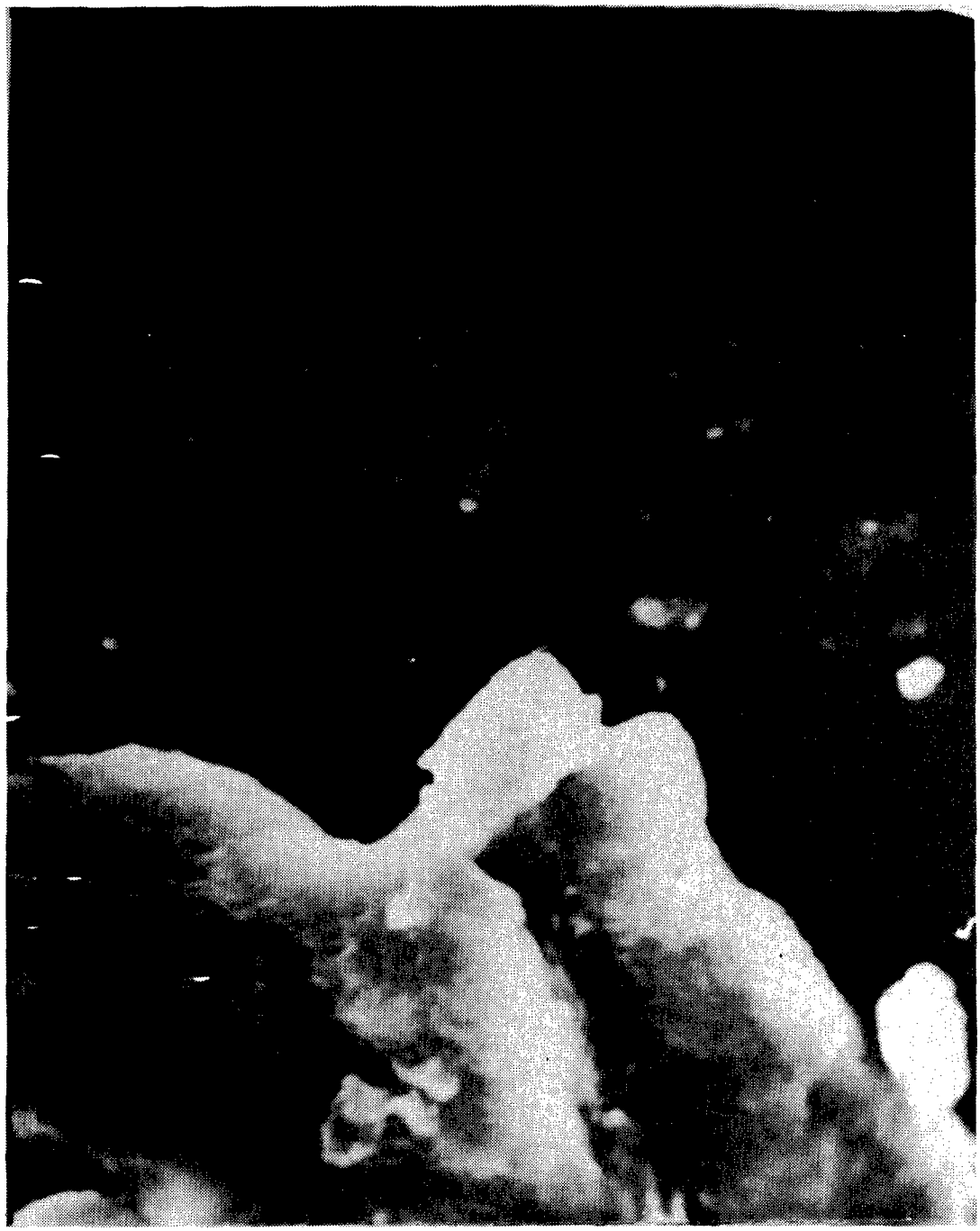


Figure 4. SEM of Stable Crack Growth Surface

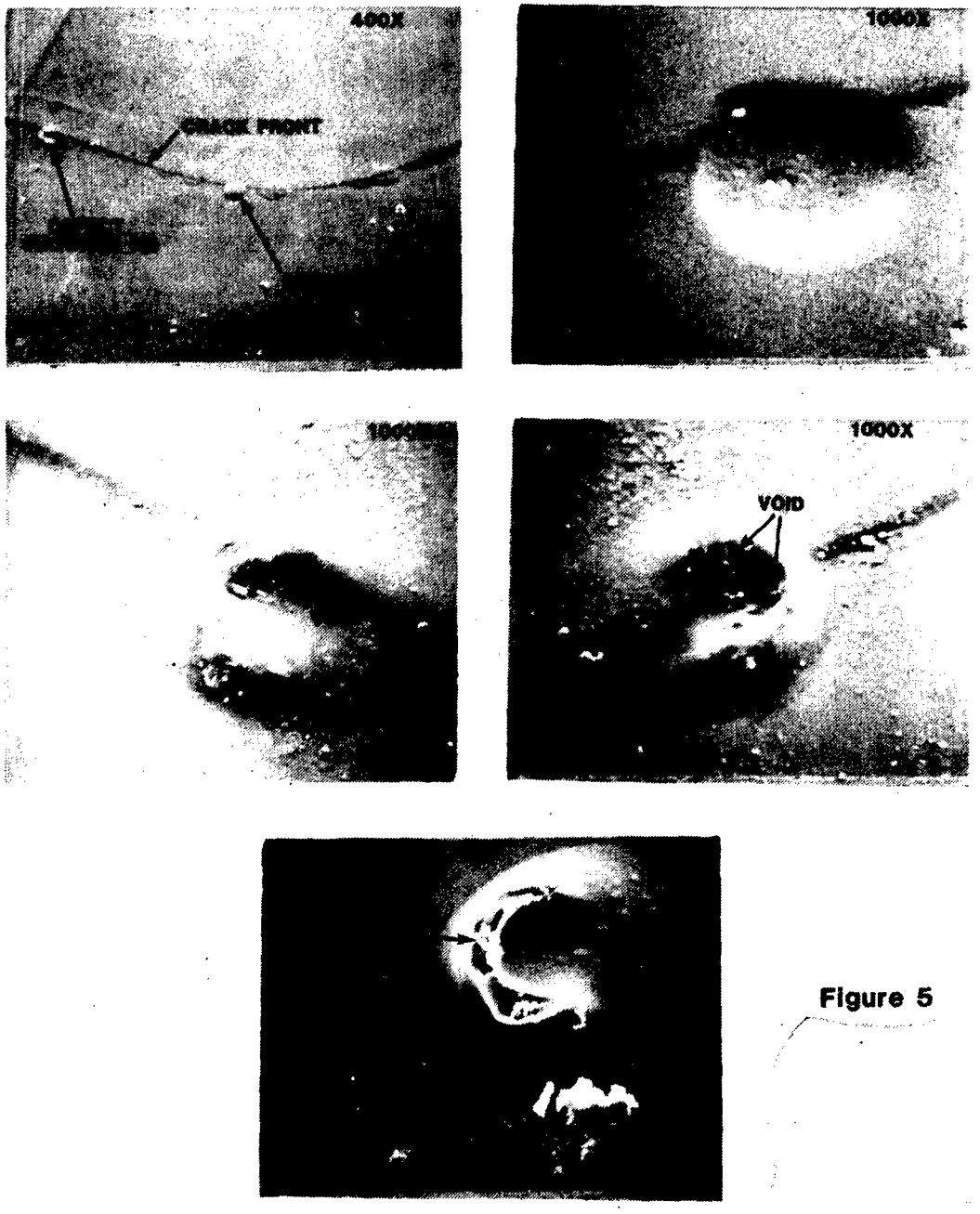


Figure 5

Figure 5. SEM of Slip Stick Surface

much larger in diameter, in the order of 2 to 5mm. In some 3501 specimens, "button" shaped chunks (flat on one side and convex on the other) would pop out from the surface of the bar. The "convex sides" all had very smooth surfaces with a distinctive navel shape dip on the newly formed surface. All these observations from earlier experiments can be explained in light of the recent findings of an Air Force sponsored contract "Processing Science of Epoxy Resin Composites" (Reference 10). The investigators of this contract found that unreacted DDS crystals are responsible for void formations in epoxy composites during the processing procedure. The defects on the crystallite will trap water which will be vaporized to form voids. If the crystallite is embedded internally in vitrified matrix at high temperature the vaporized water will create a very localized high stress region, which may swell the surface if the material is ductile or pop a "button" with a navel shaped center if the material is brittle. The domes observed here are very much similar in shape, although the CT specimens had not been subjected to similarly high temperature. The domes shown can be found on other specimens fracture surfaces which had been contaminated by human contact (wiping the surface with fingers). The origins of the formation of these domes are not clear, but moisture absorption after fracture is suspected to be the cause, and may be related to the earlier observations on epoxy bars. It should be noted these are found only on the fractured surface, not on the surfaces from the molds.

The defect shown in Figure 5a is unique to this specimen. Since this is the only room temperature "Stick-Slip" specimen of 5208, it is not sure if this feature may have been responsible for the different behavior. But both crack fronts on this specimen have such defects on them.

Figures 5c, d, and e show these defects in higher magnification. The new moon shape outline is present in all three pictures. Figure 5c and d show the defect as a cavity. All three pictures however show the presence of void sites, which may have been responsible for forming such features during the fracture process. An interesting question is whether these defects are related to the "Stick-Slip" load pattern and/or

causing the crack propagation to stop at that location, or the above mentioned factors are actually responsible for such defect formations around the pre-existed voids.

The K_Q calculations from the "Stick-Slip" pattern gave higher values. The pattern shown in Figure 3a gave values of 0.615 MPa $\sqrt{m}$ and 0.720 MPa $\sqrt{m}$; Figure 5b gave 0.490 MPa $\sqrt{m}$ comparing with the average value of 0.482 MPa $\sqrt{m}$ from other specimens. The K_Q arrest value of the "Stick-Slip" is 0.528, reasonably close to the K_Q of stable crack growth. These numbers are listed in Table 2. The data fit nicely with the results reported by Yamini and Young (Reference 7): that K_I initiation is always higher than K_I arrest, and that the K_I arrest value is about the same as that of K_I from stable crack growth.

Since we are interested in getting multiple fracture calculations from one specimen, loading other than the continuous displacement were attempted. Figure 6 shows the load curve of an intermittent displacement experiment. The specimen was loaded until fracture occurred and then the displacement was stopped to allow the arrest of the crack front. The specimen was then reloaded to initiate another fracture. The arresting load values are identical to that of subsequent initiation, supporting the interpretation that stable crack growth is a result of K_Q initiation being the same as that of K_Q arrest (Reference 7). Such a loading pattern gave more than one fracture load reading, but unfortunately, the fractured surface was as smooth as that from the continuous displacement specimens. There was no demarkation on the surface to indicate the positions of the crack front during the arrest or initiation.

In order to get information of the corresponding crack length, the specimen was partially unloaded after the fracture. The slope from the subsequent load vs displacement curve, if properly calibrated, should provide the crack length information needed for the K_Q calculation. Figure 7 shows such a load curve. It can be noted that after unloading, the load needed for fracture to re-initiate was slightly higher. This can mean that the crack tip was blunted as the result of the unloading

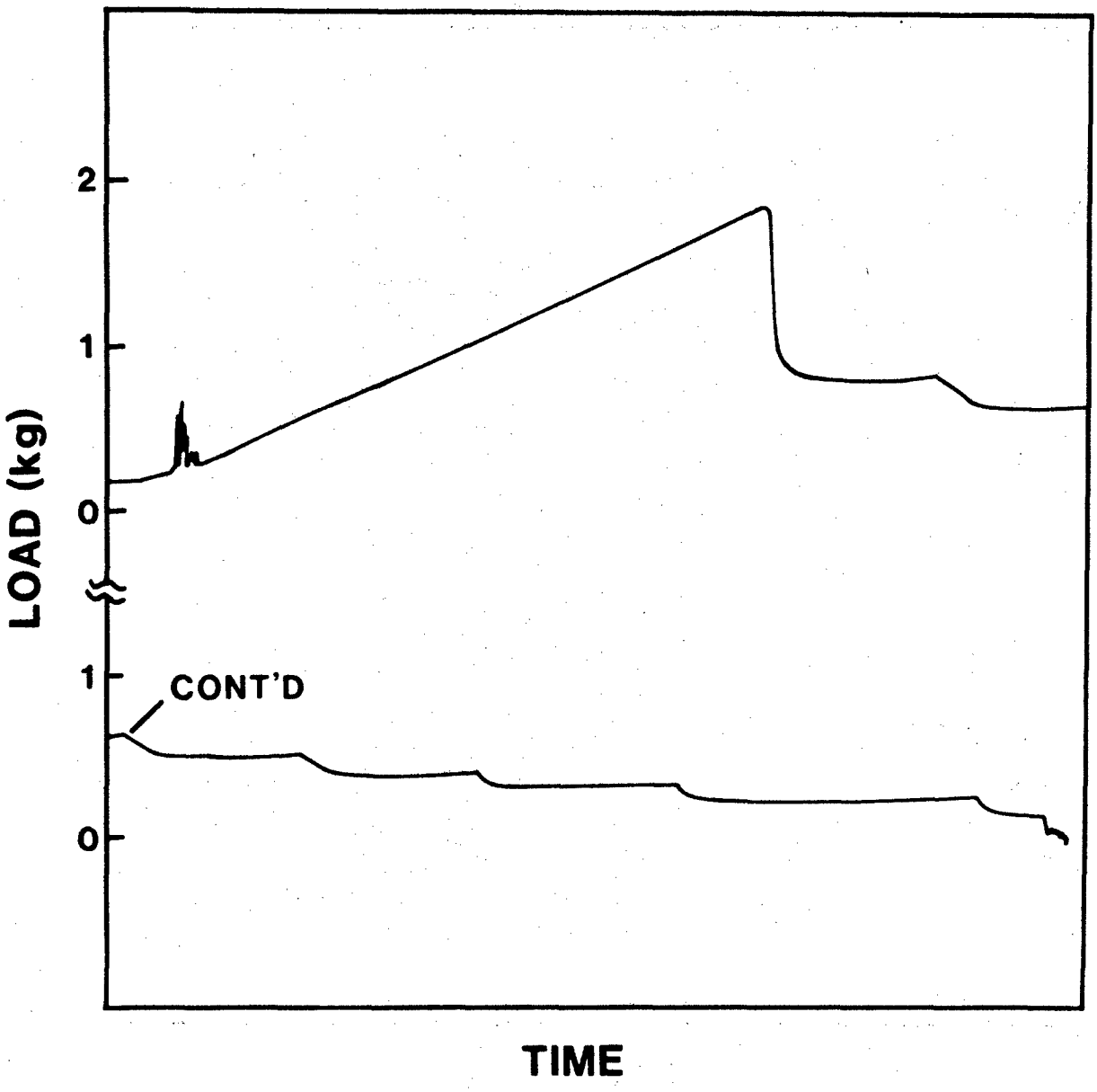


Figure 6. Load Diagram of Load-Stop Experiments

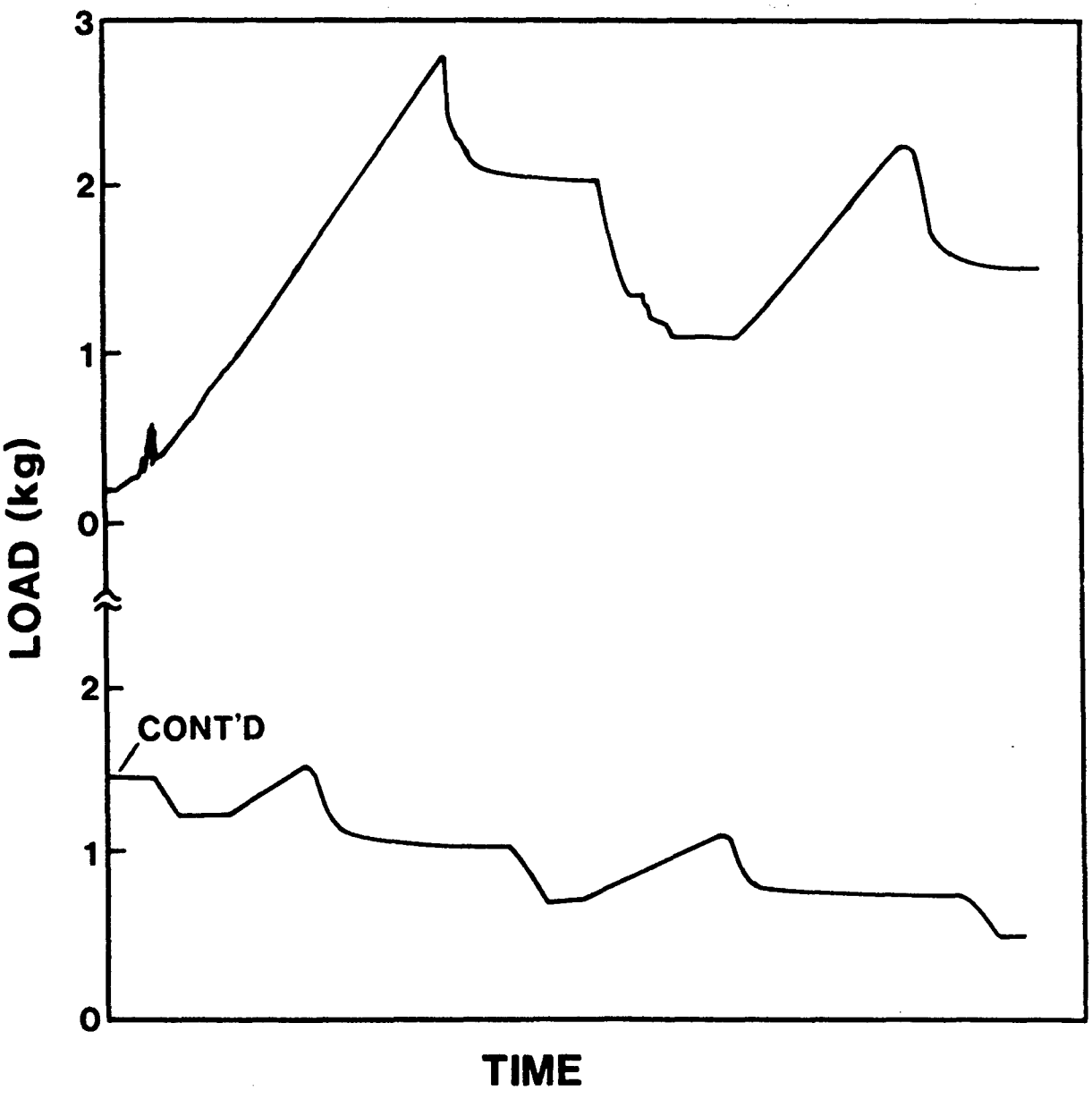


Figure 7. Load Diagram of Load-Unload Experiments

and reloading. Yet the differences were small enough that they can be conveniently ignored considering the experimental uncertainty associated with each measurement. For these epoxy specimens, clear visible marks of the crack front of each fracture initiation were present on the fractured surface, so direct measurements of the crack length can be carried out after the fracture experiment. For other materials which do not yield marks on the surface during the loading and reloading cycle, the slope measurements method will still afford an alternative in getting the crack length information. The region that was fractured with the instrument was perfectly flat and was normal to the sides of the specimen.

The fracture experiment was performed with the load - unload cycle until the crack length was too deep and the load need to re-initiate the fracture was too small for meaningful K_Q calculation. Then the specimen was separated by hands into two halves. The region that was separated by hands, however, always gave a sloped fracture surface. This fracture appearance is similar to the "Full Oblique" type described in the ASTM for metal fracture (Reference 5, Figure 12). The SEM of the specimen end is shown in Figure 8. Figure 8b showed the appearance of the "Full Oblique" feature. Figure 8a show both the flat region and the hand-torn region under higher magnification. The vast difference in appearance between the two regions is obvious.

In some instances, the razor blade initiated crack tip was not sharp enough. A blunt crack tip will always give a K_Q value that is too high. The load curve of a typical blunt crack tip specimen is shown in Figure 9. The blunt crack tip was characterized by the unstable crack propagation of the crack front of the first fracture. The crack front will travel a much longer distance than usual.

Usually several hazy bands will appear on the fracture surface associated with such an unstable crack propagation. At first these hazy bands caused much confusion about assigning crack length values for K_Q calculation. Under the microscope the appearance of these bands has misled us to interpret them to be regions where crazing have occurred.

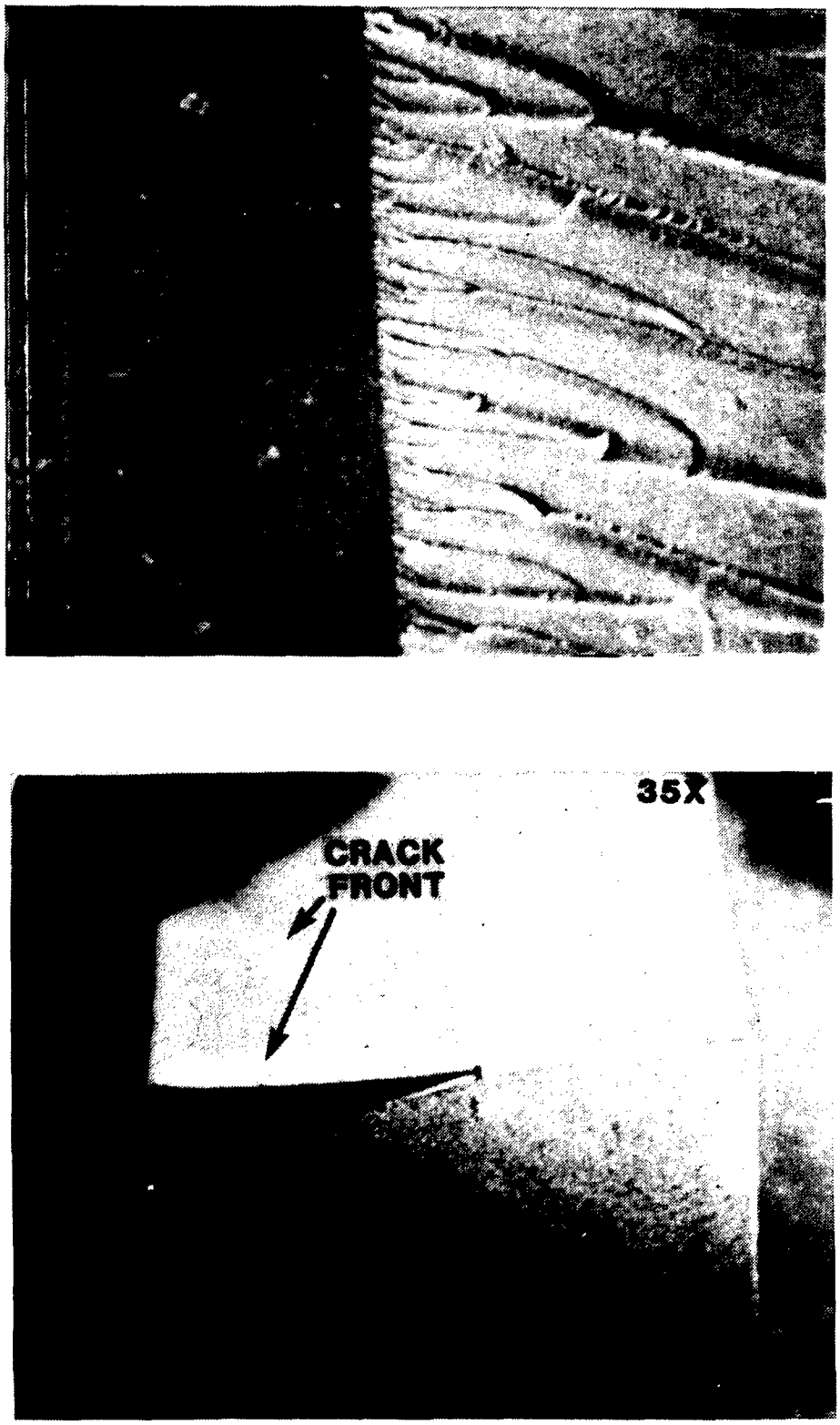


Figure 8. SEM of Specimen End Showing the Sloped Feature of the Hand-Torn Region

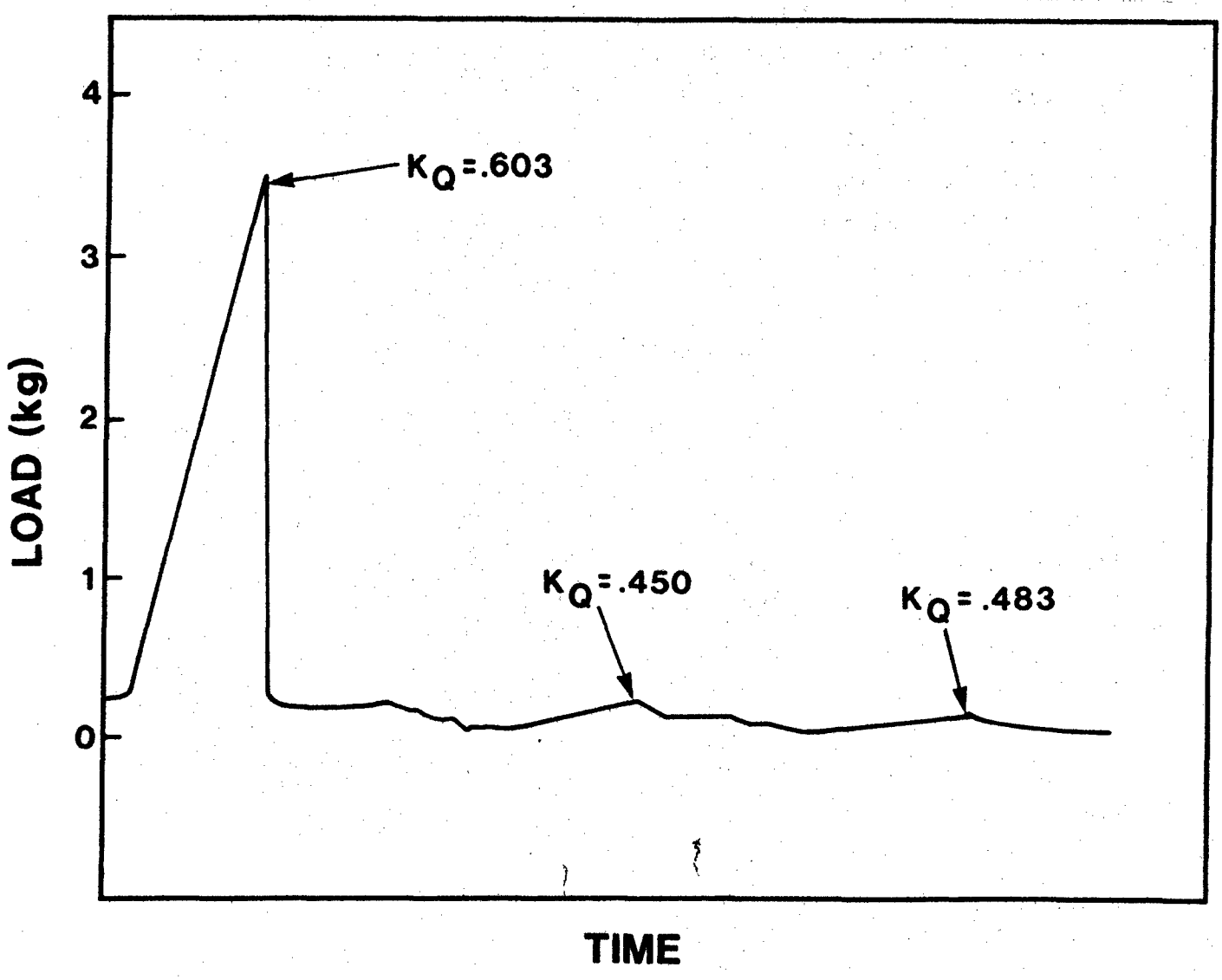


Figure 9. Load Curve from a Specimen with a Blunt Crack Tip

Under SEM, it is revealed that for most smooth fractured surfaces, there are lines running in directions normal to the crack front. The lines are very small and relatively low in density per sectional area, so in low magnification under the optical microscope, they are not visible. If we follow these lines in the same direction as the crack front propagation, these lines branch out into many lines and appear to be deeper after they run into the hazy band region. The lines in the hazy band region re-group into fewer lines as they come out of the region. This re-grouping of lines is shown in Figure 10a. The re-grouping or branching of these lines then appear to form a smooth and continuous transition front between the two regions.

These fronts were parallel to the crack fronts, which may indicate that the specimen was experiencing some changes in condition during the front propagation rather than location dependent changes in the specimen. The difference of this transition front and crack front resulted from the arrested and re-iniatiated fracture can be observed by comparing with Figure 10b. Under optical microscope, such a front can be seen as a clean line separating two mirror smooth regions. Under SEM, the line is really featureless. The two different shades of darkness separated by this line suggest that there is an abrupt change of plane angle.

The fact that the hazy bands usually are associated with the unstable crack propagation of the blunt tip which leads to higher than average K_Q may leas to the following speculation. The excess energy needed for the blunt tip to fracture resulted in two fronts of shock waves propagating away from the fracture site. These waves hit the boundaries of the specimen and were reflected back toward the plane of fracture. When they meet at the plane of fracture, the crack front region was experiencing a compressive mode which may have caused the branching of lines. A systematic study of the relationship of geometry size and the periodicity of these bands may help in explaining the origin of the hazy band.

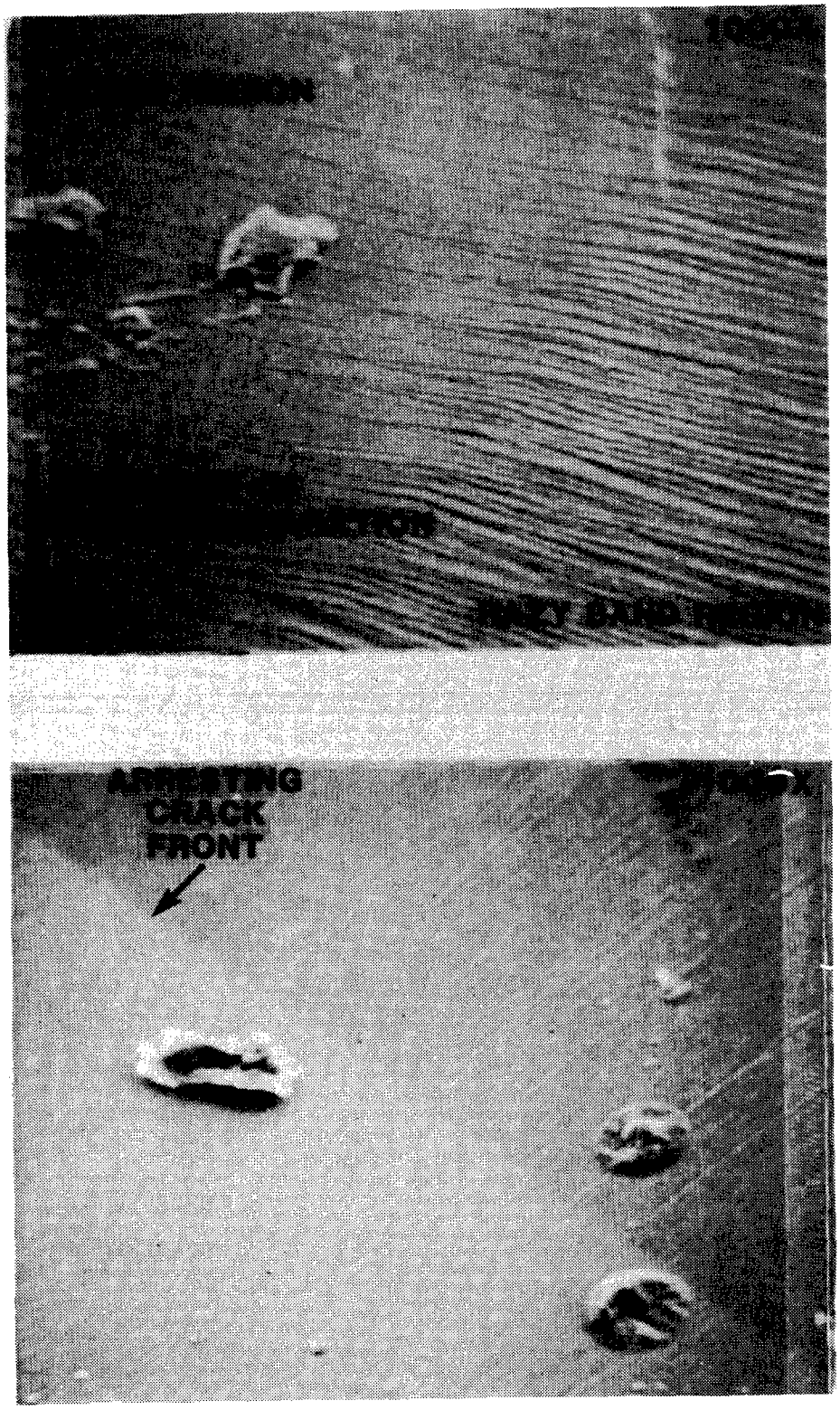


Figure 10. SEM of the Hazy Band and Arresting Crack Front

3. CRACK LENGTH MEASUREMENTS

Post test examination of the fracture surfaces showed substantial curvature of the crack initiation/arrest front. The amount of curvature appeared to vary with experimented conditions. In almost all cases, the curvature did not meet ASTM criteria, and thus the resulting data should cautiously be used for exploration purposes. It is speculated that the curvature systematically changes with test temperature, loading rate and duration due to changes in the mechanical constitutive relations and yield interaction with the finite specimen thickness. At elevated temperature tests in air, complex curvature was noted, possibly due to surface oxidation effects. We think there is fruitful information to be gained from a systematic analysis of the crack curvature data, but have assigned a moderate priority to it, and have set it aside for some later time.

Since the fronts show similar shape of curvature (Figure 2), three points can be readily identified; the front at the left edge, the front at the right edge, and the maximum of the crack length (labelled L,R and M, respectively). The crack lengths as measured from these positions were used to calculate K_Q. One specimen's data are listed in Table 1.

It can be observed from the data, that the K_Q (M) values are increasing with increasing a/W, while that of K_Q (L) & K_Q (R) are decreasing. These are shown in Figure 11. This implies that the curvature of the crack front progressively becomes deeper as it travels down the specimen.

The crack lengths at these three positions were then averaged with the equation A = (2M + L + R)/4. The A values were used to calculate K_Q (A). These values show no a/W dependency, and agrees with the values obtained from relatively flat cracks.

Similar data have been calculated using only the maximum crack length by Kenner (Reference 11). In his studies, the room temperature fracture toughness was 0.540 MPa $\cdot\sqrt{m}$ for a/W $\sim$ 0.5. This appears consistent with the K_Q value of 0.560 MPa $\cdot\sqrt{m}$ estimated for the maximum crack length at a/W 0.5 from Figure 11.

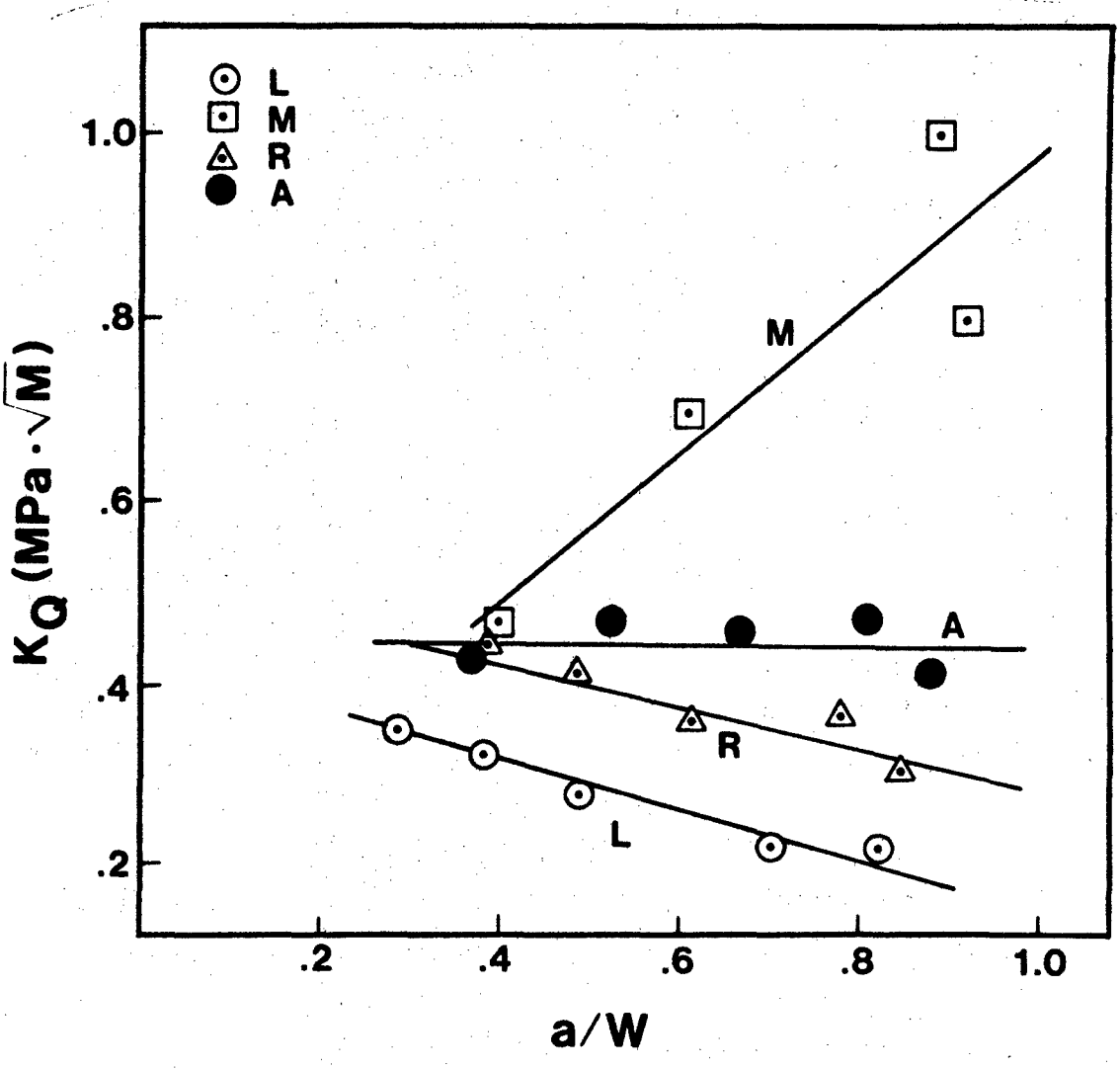


Figure 11. K_Q vs a/W Plot of a Typical 5208 Specimen

Although the curvature of the crack fronts has made most of the measurements to fail the ASTM criteria, the averaged crack length method empirically removes the curvature dependency on the K_Q and provides numbers that we can use for preliminary material comparison purposes.

4. K_Q RESULTS

Two sets of data are available. One set of data is from 3501 epoxy at ambient temperature. The crack front from these samples were all relatively straight, so the maximum crack length were used for the K_Q calculation. The experiments were in continuous displacement mode. All load curves gave the "Stick-Slip" pattern, so K_Q initiation and K_Q arrest were calculated (Table 2). The K_Q initiation (.548 MPa$\cdot\sqrt{m}$/592 psi$\cdot\sqrt{in}$) reported by Wackenhut et. al. for the same lot of resin (Reference 12). They also used the ASTM procedure E 399-78 as a guide, but used the beam specimen of 1/8" thickness instead of the CT specimen. It was not mentioned whether the load curves resembled "Stick-Slip" or stable crack growth behavior. Only one Klc value was reported.

The second set of data is that of 5208. The K_Q values are listed in Table 2 and plotted in Figure 12. All data were generated under the load-unload mode. In all cases, the force reading just before the unloading was very close to the subsequent fracture initiation force, implying the stable crack growth behavior.

The data from the high temperature runs contain two different temperature soak times (5 min and 10 min) before the fracture experiment was initiated. For the 150°C and 200°C data, the K_Q values have decreased as a result of longer soak time. For the 250°C data, the values have increased. This shows how the soak time can be used an an additional variable to monitor material properties changes. The changes observed at 200°C and 250°C can be interpreted as the results of different extent of additional cure. In light of the post cure temperature, it definitely cannot be the case for the 150°C data. It is possible that the five minutes soak time is insufficient to bring the specimen temperature to equilibrium.

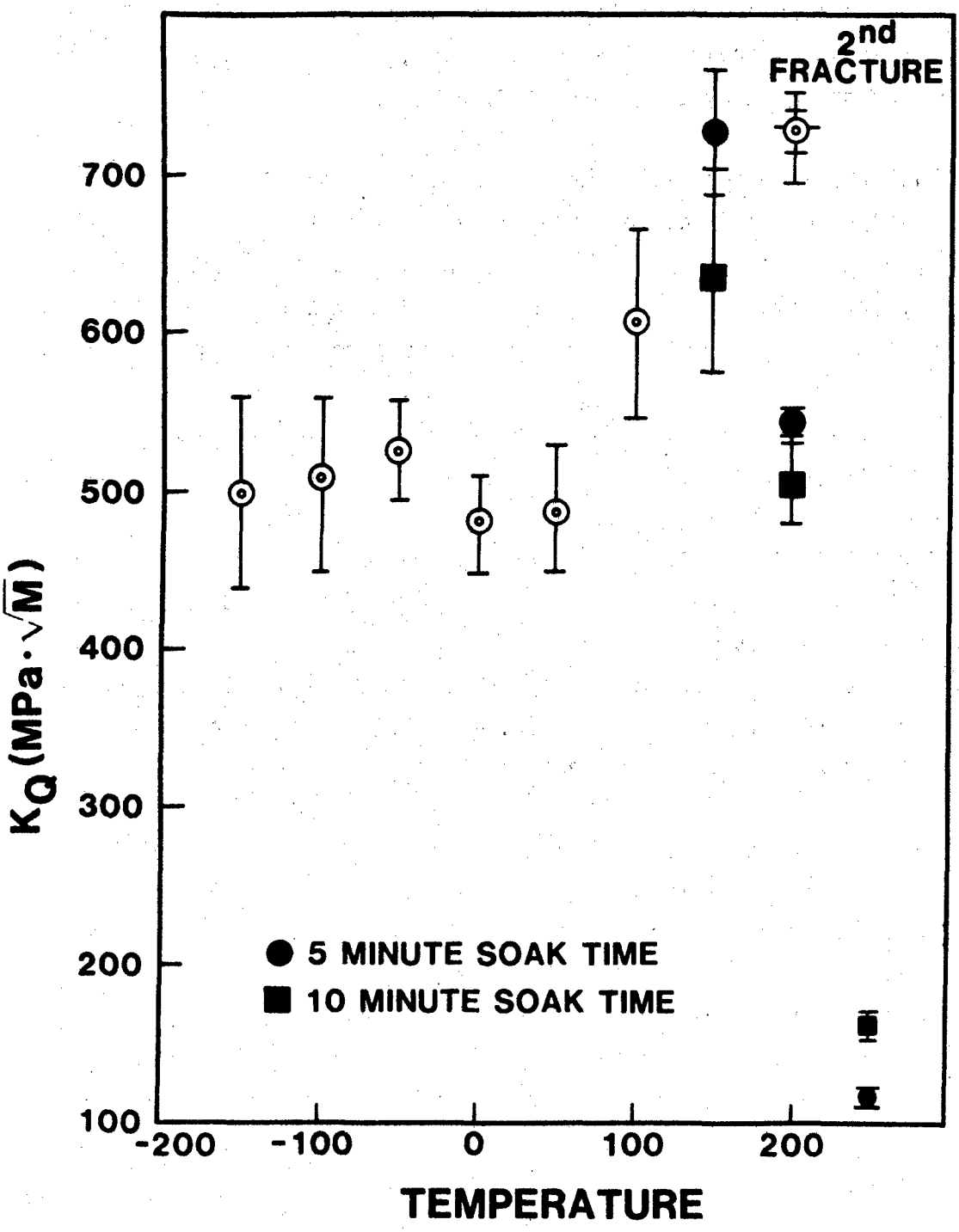


Figure 12. K_Q vs Temperature of 5208 Epoxy System

All the load curves at low temperature up to 100°C show similar characteristic as shown earlier. The 150°C, 200°C, and 250°C load curves are different and they are shown in Figure 13.

Without exception, all specimens at 200°C (both 5 and 10 minutes soak time) gave two fracture peaks. The first peak always showed a slow crack growth region after the displacement was stopped. After the unloading, the second fracture resulted in a fast crack growth which propagated the front too far down the sample to permit another measurement.

The K_Q values from these two peaks are clearly different (Table 2) The first peak value decreases with increasing soak time but the second value remains unchanged. The fracture surfaces of these specimens clearly show two distinct regions. The first region extended from the razor crack front to the second front. The feature of this region is very rough and the SEM is shown in Figure 14a. The second region is mirror smooth and extends to the end of the sample. Ocassionally hazy bands are observed in this region, but they are usually not as distinct as those that were observed from lower temperature experiments.

Based on these results, it is possible that the first fracture was a result of the fine crack initiated by the razor blade. Because of the temperature characteristic of the material, the specimen released the load by propagating the crack front slowly even though the displacement had stopped. This slow crack growth left a very rough surface, and as a result, the crack front became blunt for the second fracture. The excess energy needed to initiate the second fracture then caused the unstable propagation of the crack front, and gave a higher calculated K_Q value.

All the specimens at 150°C gave catastrophic fracture load curves so only one K_Q can be calculated from each specimen. The fractured surfaces are all smooth with the presence of the hazy bands. It is not sure if this is characteristic of the material at this temperature or just the result of blunt initiated crack fronts. The later explanation

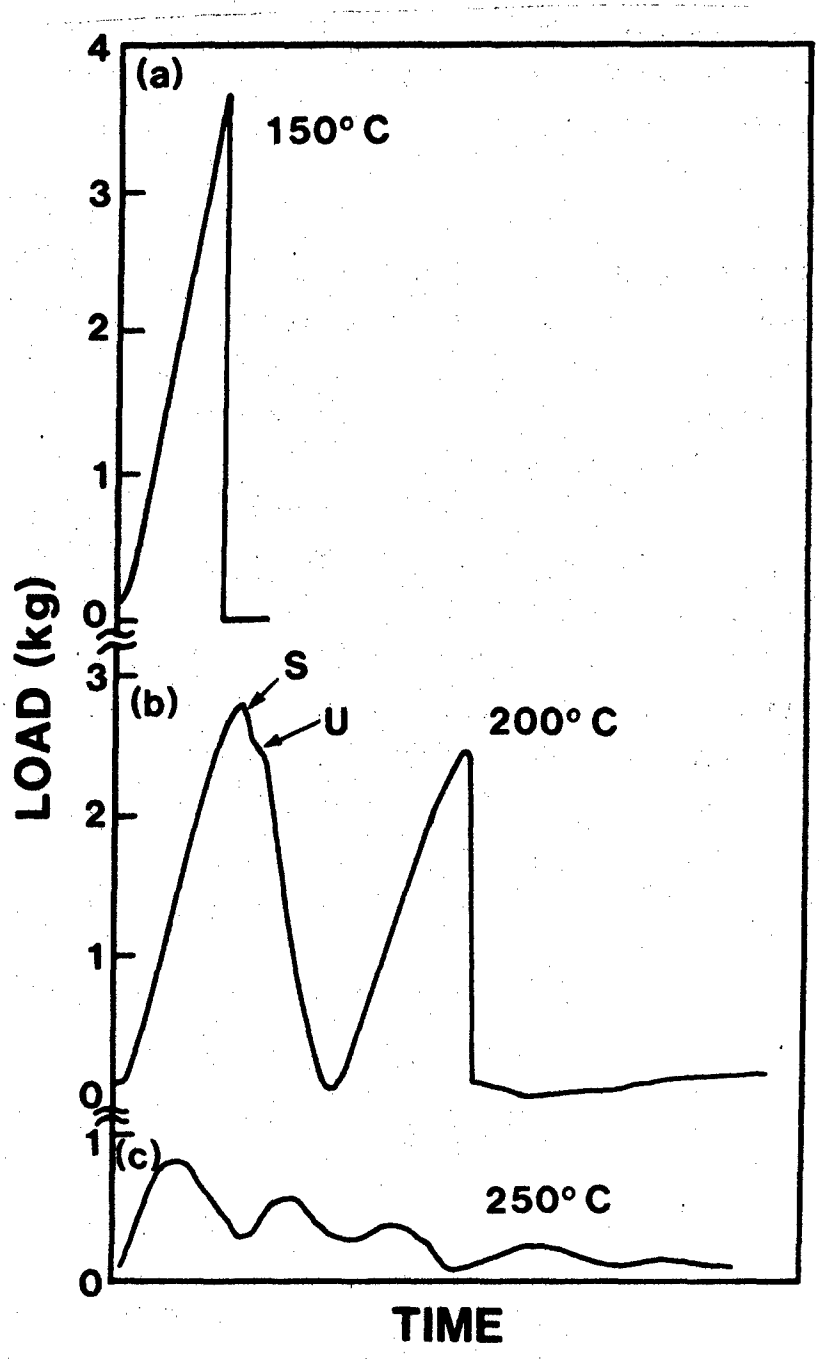


Figure 13. Load Curves from Different Temperature Fractures

can be plausible because we have experience at room temperature experiments where one operator can very consistently produce crack tips of the same degree of bluntness, generating statistically very consistent but erroneous, results.

The load curves at 250°C also show the stable crack growth after displacement was stopped, although no rough regions were observed on the fracture surfaces. The succeeding fractures all gave similar K_Q values. The regions between two crack fronts is shown in Figure 14b. The maximum loads were used for the K_Q calculations.

All these data were generated with the intention to test the different temperature experimental procedure. Although interesting results were obtained, the study is not considered complete, and there remain several unanswered questions. Additional experiments with systematic variation of parameters are recommended, with appropriate consideration of the material's time dependence to fully characterize the high temperature fracture behavior of this epoxy system.

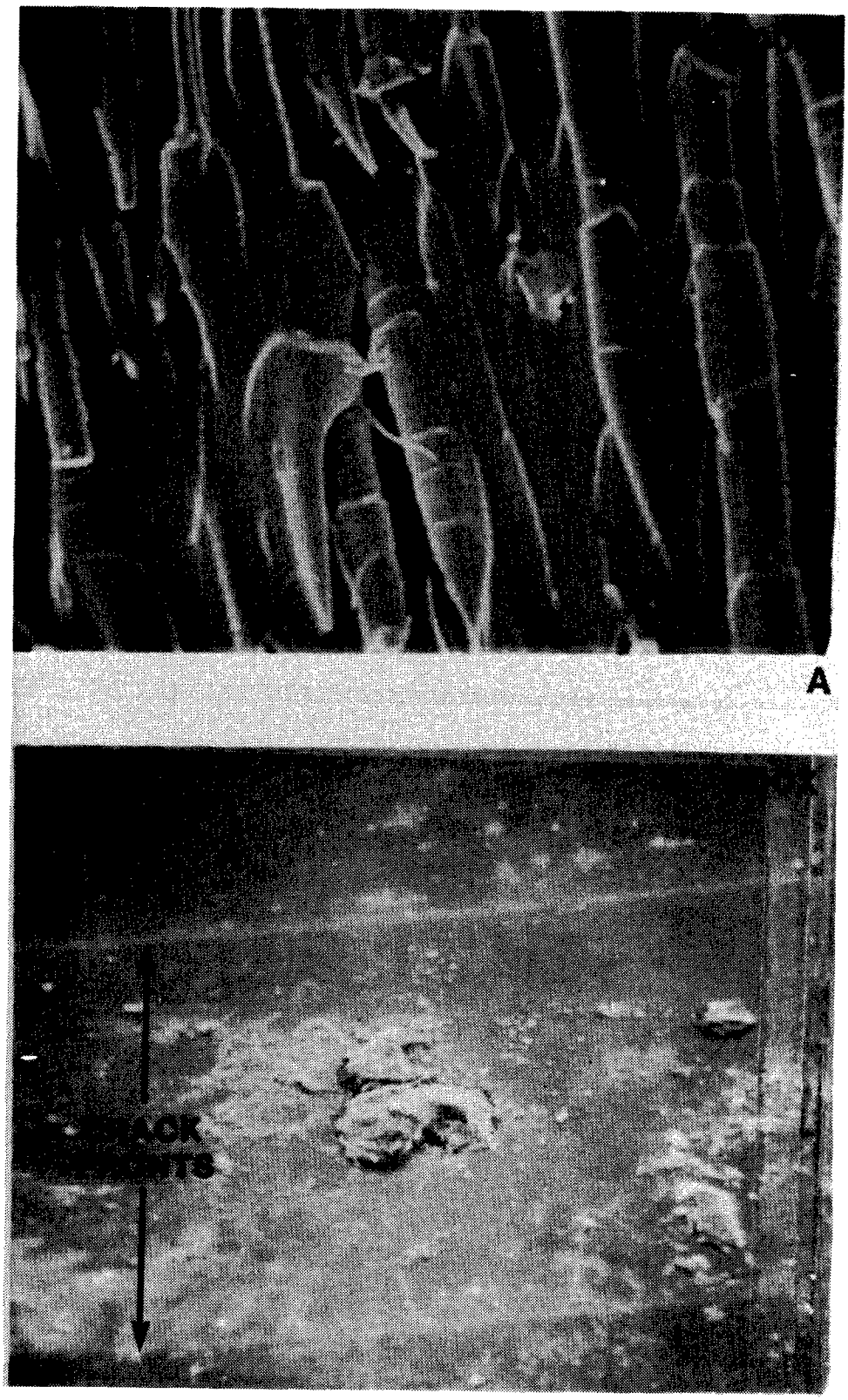


Figure 14. Fracture Surfaces of the Specimen from an Experiment @200°C

CONCLUSION

This work has accomplished the goal stated in the milestone. The work has developed a feasible experimental procedure to test fracture initiation behavior using a Rheometrics Mechanical Spectrometer (RMS). The reason for using the RMS is to capitalize its unique temperature and environmental capability, which not only covers a wide temperature range, but also gives very reproducible heating and cooling profiles in a controlled nitrogen or air environment. Interesting results have been obtained even within the scope of the current milestone, which reveal new vistas for further in-depth investigations. An area of special interest is in the time-dependent fracture behavior of polymers at high temperatures.

Two problem areas were encountered in this study. First, the compliance of the mechanical converter seriously limits the application of this procedure with the RMS to test materials much tougher than the current epoxy systems. An alternative would be to use a stiffer test machine. A newly designed Instron environmental chamber is now on order. This chamber should have similar temperature capability as that of the RMS, and the Instron will be a stiffer loading device, and will thereby avoid the compliance problem.

Secondly, with the procedure and sample geometry. K_Q values relating to the fracture process can be generated. Whether the value obtained is truly the Klc value or not will depend on each material tested. Additional tests for each material will have to be performed to ascertain that the plane strain condition is satisfied. Such an effort is currently underway for the 5208 epoxy resin. In spite of this shortcoming, the small sample size requirement with this CT geometry makes it feasible to incorporate the testing in the 50 gram evaluation scheme, which will provide valuable preliminary material properties in resin development programs.

TABLE 1

RESULTS FROM A TYPICAL 5208 SPECIMEN$^{\Delta}$

a/W(L)	a/W(M)	a/W(R)	a/W*(A)	K_Q(L)	K_Q(M)	K_Q(R)	K_Q(A)	
K_Q and a/W from one specimen								
.376	.394	.295	.365	.450	.472	.365	.437	
.388	.608	.486	.523	.324	.648	.425	.477	
.545	.754	.616	.667	.284	.750	.370	.461	
.700	.882	.781	.811	.228	1.003	.378	.480	
.821	.919	.849	.876	.237	.805	.309	.423	
			Mean	.305	.736	.369	.456	
			Standard Deviation	.090	.196	.041	.025	
Averaged K_Q from five CT specimens (FA04A07A-8)								
				.364	.914	.324	.480	(4)$^{+}$
				.257	1.092	.287	.466	(2)
				.305	.736	.369	.456	(5)
				.343	.860	.360	.493	(4)
				.310	.902	.404	.480	(3)
			Mean	.322	.870	.354	.475	
			Standard Deviation	.034	.108	.035	.014	

* $a/W(A) = (a/W(R) + a/W(L) + 2a/W(M))/4$

+ Number of K_Q calculation from each specimen

Δ All K_Q values in units of MPa $\cdot\sqrt{m}$

TABLE 2

K_Q RESULTS OF 3501 AND 5208 EPOXY SYSTEMS

EPOXY SYSTEM	TEMPERATURE (°C)	K_Q (MPa·√m)	
3501	RT	.548 ± .039	(i)
		.372 ± .032	(a)
5208	RT*	.667	(i)
		.528	(a)
	-150	.500 ± .060	
	-100	.510 ± .050	
	- 50	.524 ± .030	
	0	.481 ± .032	
	RT	.482 ± .010	
	50	.487 ± .040	
	100	.605 ± .060	
	150 (5 min.)	.728 ± .034	
	(10 min.)	.636 ± .063	
	200 (5 min.)	.540 ± .012	
		.727 ± .018	(1)
	(10 min.)	.506 ± .025	
		.726 ± .027	(1)
	250 (5 min.)	.119 ± .005	
	(10 min.)	.161 ± .009	

*From one continuous loading experiment

i - initiation

a - arrest

1 - from 2nd fracture

APPENDIX

PROCEDURE FOR RMS FRACTURE ANALYSIS W/MULTI-TEMPERATURE RUNS

1. Set-up:

   a. Put on Air Bearing Transducer (see instructions in notebook).

   b. Make sure the Air Brake is on! Turn on compressed air supply.

   c. On the RMS:

      (1) MAINPOWER: ON

      (2) DRIVE: OFF

      (3) MOTOR: BRAKE

      (4) ROTATIONAL SPEED: As specified by the experiment

2. Mounting of Environmental Chamber onto the RMS:

   a. The medium size chamber is used.

   b. Line up the large rod on the RMS with the hole on the Environmental Chamber and slide the chamber onto the rod (hold all attached wiring to the chamber with your left hand).

   c. Slide the back plate of the Environmental Chamber so that its four outer screw holes line up with the screw holes on the RMS.

   d. Insert the screws and tighten.

   e. Connect the wires in the following manner:

      (1) The Thermocouple wire (colored, smaller diameter) is connected to the FEMALE connector of the Digital readout (located on the upper RIGHT of the RMS, next to the upper external switch).

      (2) The Gray wire, labelled CHAMBER, is connected up to the CHAMBER TC connector (located on LEFT side of the RMS below the Environmental Control Box).

(3) The Gray wire, labelled PRT is connected to the PRT connector (located in back of the Environmental Control Box).

3. Hook-up for Hewlett-Packard 7046A X/Y Recorder:

a. Connect the Recorder to the Control Panel:

(1) The input of the Y-1 section of the Recorder is connected to a hookup in back of the Rheometrics Signal Conditioner, marked RECORDER (BLACK Hookup, NOT RED).

(2) The input of the Y-2 section of the Recorder (2 RED-RINGED FEMALE BANANA Connectors) is connected to the Angular Position Control Output (BNC Connector) located on the Front of the Rheometrics Signal Conditioner.

NOTE: In both a and b use Banana Connectors w/the Ground plugged into the LOW socket!!!!!

b. The range of the Y-1 on the Recorder is set to 20 mV/in.

c. The range of the Y-2 on the Recorder is set to 5 V/in.

d. The X axis is set on TIME BASE with the appropriate time scale (as specified by the experiment).

e. Turn the Recorder on:

(1) LINE: ON

(2) SERVO: ON(Then STANDBY)

f. Insert Chart Pens:

(1) The BLUE pen is inserted into pen holder CLOSEST to the bar. This will measure the Transducer's Z-FORCE.

(2) The RED pen is inserted into the pen holder FARTHEST away from the bar. This pen measures the travel in radians.

g. Insert Chart paper:

(1) Make sure the paper is square against the bottom of the Recorder's chart surface.

(2) Turn CHART switch to HOLD

(3) Smooth the paper out.

4. Calibration of the Recorder:

a. On the control panel of the Signal Conditioner:

(1) Z-FORCE: 5,000 grams

(2) Press zero button on control panel, and use recorder zero dial (Y-1) to position blue point at 5 inch mark of the paper.

b. Release zero button and, use the Z-FORCE COARSE and FINE ADJUSTMENT to ZERO the Y-AXIS, again at five inch mark.

c. Put the 1,000 gram weight on the bottom stage and you should see a two inch deflection on the chart. If the deflection is not EXACTLY two inch use the CAL knob on top of the Y-1 RANGE selector to get the two inch deflection (ONCE THIS HAS BEEN SET, DO NOT TOUCH THE CAL KNOB AGAIN UNTIL THE EXPERIMENT HAS BEEN COMPLETED).

d. Take off the 1,000 gram weight and the REPEAT Step 2 and 3 to proper calibration.

5. Mounting of the Fracture Fixtures:

a. Press external input to zero position control. The digital display on the signal conditioner should read approximately 0.000.

b. Turn off the external input. Turn the upper spindle to increase angular position digital display. Stop at the FIRST 1.200 ±0.005 reading.

c. Set a gap of 1.2 cm between the upper tool assembly and the fixture. Carefully mount the upper fixture, making sure to run thermocouple through center of the fixture and LINE UP GUIDE PIN on RMS WITH APPROPRIATE HOLE on FIXTURE! The gap should not be changed during the mounting.

d. Tighten knurled ring and the top lock ring to hold the upper fixture WITHOUT DISTURBING the 1.2 READING on the ANGULAR CONTROL.

e. Mount the bottom fixture and tighten the lock-ring w/the tools.

f. Readjust the Z-FORCE (fine Adjustment) to "Zero" the Recorder. There will be some deflection on the Recorder due to the mounting of the fracture fixture.

g. Reposition Y-1 zero to 2 inch mark on chart paper (repeat IV-1b).

h. Lower the top fixture by turning the knurled know on the side of the RMS "CW" to actually lower the fixture. DO NOT LET THE FIXTURES TOUCH!!!!!!!! May have to adjust the gap stop to do this (the knurled knob next to the Gap Indicator).

i. Insert the Aluminum bar standard between the fixtures and tighten the Air Brake (very tight).

j. Remove the Aluminum bar standard.

6. Turning on of the Environmental Chamber

a. Check liquid nitrogen reservoir to make sure there's no water!!

b. Environmental Control Boxes (If nitrogen environment is desired):

(1) Toggle switch to LIQUID
(2) Toggle switch to AUTO

c. Temperature Programmer Panel:

(1) Sweep toggle: UP
(2) Toggle switch to LOCAL
(3) Toggle switch to LOW TEMPERATURE
(4) HIGH SETTING: 675K
(5) LOW SETTING: Dial the setting to Room Temperature initially.

d. RMS Controls:

(1) Press in the BLOWER button and this will start the liquid N_2 (on the Environmental Control Box, the LOW LEVEL light will go on).

(2) When the SOLENOID VALVE is ACTUATED to INTERMITTENT on/off, press the HEATER/BLOWER BUTTON.

(3) The low temperature setting, initially set to room temperature, can be dialed to desired temperature.

7. The Initial Razor fracture in the sample can be induced by one of two methods.

a. Running a razor blade across the slot

(1) Firmly clamp fracture sample across the width just below the slot, with the slot at the top.

(2) Using a fresh razor blade, run the length of the blade across the slot applying slight pressure to the razor blade. Be sure blade is perfectly horizontal as initiating the crack.

(3) Check the sample for crack, if no initial crack is seen repeat steps a and b.

b. "Tapping" razor blade in slot.

(1) Firmly clamp the fracture sample across the width just below the slot, the slot would be on the top.

(2) Insert a fresh razor blade into the slot, and with a "tapper" lightly hit the razor blade. Be sure blade is perfectly horizontal when initiating the crack.

(3) Check the sample for crack, if no initial crack is seen repeat steps a and b.

c. Use either method to initiate crack, just be consistent with the method used.

8. Insertion of the Sample:

a. IF THE TIMER IS NEEDED:

(1) Power toggle: ON
(2) Set A: As specified
(3) Set B: As desired (the time must be greater than that of A).

b. BE SURE EVERYTHING IS READY. Check the recorders' Y-1, Y-2 and time scale setting.

c. Check Rotational Speed Setting. The lower dial sets the pre-scaled numbers, the upper switch sets the multiplier.

d. Open the oven door/and place the sample between the fracture fixtures, inserting the pins through the holes in the sample and stages. The TOP pin should be inserted first.

(1) If the bottom pin doesn't readily go through the sample, the gap stop can be used to raise the fixture slightly and the knurled dial on the right side of the RMS may be used to lower the fixture slightly (the gap stop may have to be released before lowering fixture).

e. Close the oven door (keeping the door open too long will cause the oven to overheat, and thus shutting down the RMS). START TIMER IF DESIRED.

f. Place the sample under tension, as will be indicated on the chart when the BLUE pen goes up. (Do NOT put too much tension on the sample!!!!!) This is done by turning the knurled knob next to the Gap Indicator CCW (This will raise the upper fixture).

9. Experimental RUN

a. When the "settling" time for the sample is reached:

(1) On the RMS-DRIVE: ON
(2) On the Chart Recorder-Press the START (on the left).
(3) On the RMS-Turn the dial from BRAKE to CW.

b. When the sample has fractured, IMMEDIATELY turn the dial on the RMS to BRAKE

c. Unload the tension on the sample by turning the large knurled knob (immediately above the environmental chamber) CCW until the BLUE pen has reached its original "0" starting position.

d. Repeat steps 1c, 2 and 3 until the sample has been fractured completely through. May have to reset and start chart recorder during the run.

10. Removal of the Sample:

a. Chart Recorder:

(1) Press RESET
(2) SERVO: STANDBY
(3) CHART: RELEASE
(4) Slide the paper off

b. Turn dial on the RMS to BRAKE

c. Turn the knurled knob on the RMS (same one as in IX, 4) until the Digital readout on the signal conditioner read + 1.000 ± .005 (make sure it is the same 1.000 reading position when you start. There are at least 6 similar positions).

d. Open the oven door and take out the pins and remove the sample (this must be done fairly quickly).

e. Close the Oven door.

11. Additional runs at the same temperature or at a different temperature:

a. If a different setting is needed, dial in the desired temp. in the LOW SETTING on the Temperature Programmer panel.

b. Chart Recorder

(1) Put paper on the Recorder

(2) CHART: HOLD

(3) SERVO: ON

c. Re-zero the chart recorder, if necessary, with the Z-Force Fine Adjustment.

d. Repeat Procedure from VIII to start another run.

12. Shut down:

a. Timer

(1) Power Toggle: OFF

b. RMS:

(1) Environmental Chamber: OFF

(2) DRIVE: OFF

(3) Release the Air Brake and take off the Fracture Fixtures with the tools.

c. Make sure Air Brake is on! Turn off the Compressed Air Valve.

d. Disconnect the Chart Recorder and put it back in its plastic cover.

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